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Cyclic properties of thermal storage/discharge for Al-Si alloy in vacuum for solar thermochemical fuel production

N. Gokon[a,]*, S. Nakamura[b], T. Yamaguchi[b], T. Kodama[b]

[a]*Center for Transdisciplinary Research, Niigata University, 8050 Ikarashi 2-nocho, Nishi-ku, Niigata 950-2181, JAPAN*
[b]*Dept. of Chemistry & Chemical Engineering, Faculty of Engineering, Niigata University, 8050 Ikarashi 2-nocho, Nishi-ku, Niigata 950-2181, JAPAN*

Abstract

Thermal energy storage using phase-change materials (PCM) can be utilized for load shaving or peak load shifting when coupled to a solar thermochemical reactor, reformer, or gasifier for the production of solar fuel. The PCM is embedded in packages or used in bulk in these storage systems, and therefore the compatibility of the encapsulation materials and the selection of the PCM are key factors for ensuring the long operational life of the system. Various kinds of molten fluoride, chloride and carbonate salts, and mixed molten salt, which function at high temperatures of over 500 °C, are known to cause corrosion or thermal degradation. It is therefore worth studying new high-temperature PCM thermal storage alternatives to these molten salts for use in solar thermochemical processes. In this study, the focus was on aluminum-silicon alloy (Al-Si alloy) as a high-temperature PCM thermal storage medium, and the compatibility of this alloy with graphite-carbon encapsulation material was experimentally examined. The cyclic properties of thermal storage/discharge for Al-Si alloy as a latent-heat energy storage material was studied with respect to various thermal cycles. A thermal stability test was performed for the Al-20wt%Si, Al-25wt%Si, Al-30wt%Si, and Al-35wt%Si alloys placed in the graphite container in vacuum. The temperature increasing and cooling performances of the Al-Si alloy were measured during the thermal storage (heat-charge) mode and during the cooling (heat-discharge) mode. The oxidation level of the Al-Si alloy after the cyclic reaction (20 cycles) was evaluated using an electron probe microanalyzer (EPMA).

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Keywords: Phase-change material; Thermal storage; Aluminum-silicon alloy; Solar reactor; Thermochemical fuel production

* Corresponding author. Tel.: +81-25-262-6820; fax: +81-25-262-6820.
E-mail address: ngokon@eng.niigata-u.ac.jp

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1. Introduction

Concentrated solar radiation has specific properties, such as high density, heterogeneous distribution of thermal flux, and frequent thermal transients, because of the fluctuating insolation resulting from the passage of cloud. A solar receiver for concentrated solar power (CSP) and a solar chemical reactor for solar hydrogen production must therefore receive the concentrated solar radiation effectively and convert the high-temperature solar heat into chemical fuels by means of endothermic thermochemical processes. Examples of these processes are the two-step water-splitting cycle, methane reforming, and the gasification of carbonous materials.

Typical thermal energy storage (TES) systems contain the sensible heat of inorganic salts, in particular a binary mixture of sodium and potassium nitrates. Inorganic salts were already considered as materials for TES in the mid-1970s [1-3]. These salts are still being researched [4-7], where the main goal is to extend the working temperature range by either increasing the decomposition temperature or decreasing the melting point. Another possibility is to store the energy in the form of latent heat using first-order phase transformation, in particular the solid-solid, solid-liquid, and liquid-gas phase transitions. However, most studies focus on the solid-liquid phase transitions because of their higher energy density. The latent heat TES, as a technique in matching energy supply and demand, plays an important role with regard to thermal efficiency and economic feasibility. In comparison with sensible TES systems, the use of latent TES systems present advantages such as working within a narrow temperature range and allowing the design of smaller storage capacity for CSP. Moreover, latent heat TES systems can function coupled to a solar chemical reactor, where the endothermic chemical reaction proceeds through the use of high-temperature solar heat as an energy source. If the functioning temperature of the solar chemical reactor is compatible with the melting point of the TES material, a large amount of latent heat released at the temperatures can be used as an energy source to drive an endothermic chemical reaction in a solar chemical reactor.

A comparative study was carried out on other energy storage materials as phase-change materials in latent heat TES. Molten salts such as nitric acid, fluoride, chloride, and carbonate, as well as mixed molten salts were selected and studied as thermal storage media. The reason for this selection was their suitable phase-change temperature, high latent heat density and good thermal reliability for solar power generation and solar thermochemical process upgrading fossil fuels through endothermic chemical reaction. Fluoride molten salts have high latent heat at the phase-change temperature and sensible heat attributable to temperature dependence of a large heat capacity C_p. However, fluoride molten salts at high temperatures are strongly corrosive to PCM containers such as low carbon steel and stainless steel, as are chloride molten salts at high temperatures to metallic PCM containers. The present authors have focused their study on carbonate molten salt as a latent heat TES for solar thermochemical reformers. The reasons why this particular molten salt was selected are as follows: 1) it is relatively mildly corrosive in comparison with the fluorite and chloride molten salts; 2) it has a relatively large heat capacity C_p and large latent heat when solidifying or melting at the phase-change temperature [8-15].

The present authors have developed a tubular reactor system, using novel “double-walled” reactor tubes, with carbonate molten-salt thermal storage as PCM for the solar reforming of natural gas [8-15]. In this reactor system, an internal catalyst tube was installed in an exterior tube filled with the molten-salt and, during solar radiation, heat was conducted through the container walls and a layer of relatively low-thermal conductivity PCM to reach the catalyst and reactant gases in the internal tube. Alternatively, the reactor tube system with PCM thermal storage could be applied to a tubular solar receiver by changing the catalyst bed and passing the gas into the ceramic particle bed for heat transfer and to the air for heat transfer fluid respectively. In both instances, the high heat capacity and large latent heat of the PCM phase circumvent the rapid temperature change of the reactor tubes at high temperatures under fluctuating insolation. Because of the very low thermal conductivity of Na_2CO_3 as a thermal storage medium and the suppression of the convective flow inside the container of the reactor to delay corrosion of the PCM container, Na_2CO_3/MgO composite material (MgO ceramic particles mixed with Na_2CO_3) was tested as a thermal storage medium for the double-walled reactor tubes.

The advantages and disadvantages of carbonate molten salt for use in the reactor tubes were summarized and listed in Figure 1. As regards the Na_2CO_3/MgO composite, it has a high phase-change temperature of about 850 °C and large latent heat of about 300 kJ/kg. Therefore, the reactor tubes filled with the thermal storage material can discharge latent heat from the molten Na_2CO_3 at that temperature (850 °C), and supply the endothermic heat of the reforming reaction occurring with the methane conversion at the level of > 90% [15]. However, the disadvantages of the Na_2CO_3/MgO composite as well as Na_2CO_3 molten salt include deficient thermal conductivity and corrosion of stainless steel.

In this paper, the authors focused on metal alloy as a new latent heat TES and alternative to the Na_2CO_3/MgO composite. The cyclic properties of the thermal storage/discharge of the Al-Si alloy were examined in vacuum to evaluate the alloy as a potential PCM thermal storage material. The temperature increasing performance of the Al-Si alloy was measured during the thermal storage (heat-charge) mode and during the cooling (heat-discharge) mode. The level of oxidation of the Al-Si alloy after the cyclic reaction (20 cycles) was evaluated by using an electron probe microanalyzer (EPMA).

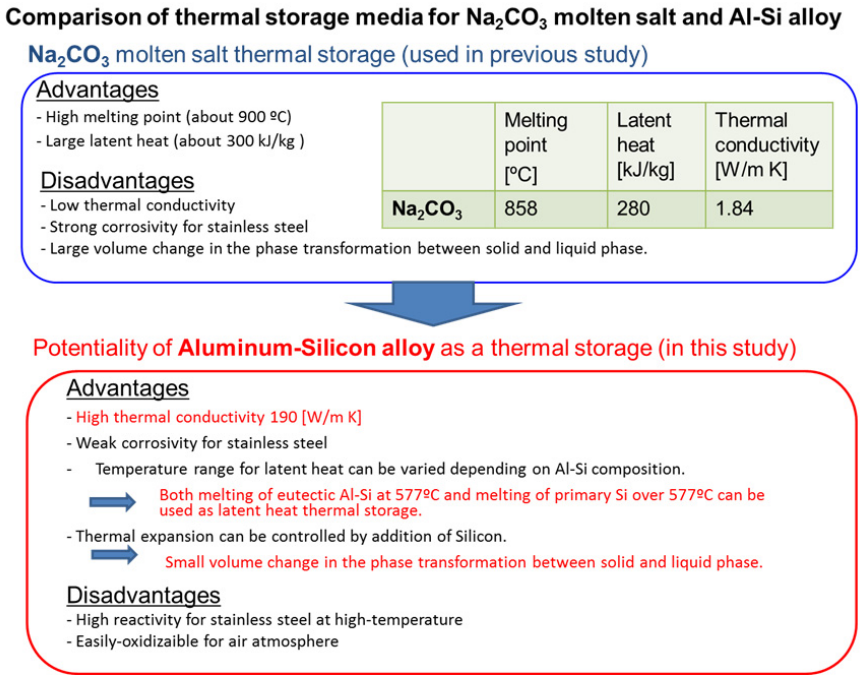

Figure 1. comparison of thermal storage media for Na_2CO_3 molten salt and Al-Si alloy.

2. Potential of metal alloys as a thermal storage medium for solar thermochemical processes

Metal alloy PCMs were proposed as TES materials more than three decades ago [16], and have been experimentally studied to some extent for TES applications [17-19]. A superior PCM must meet the following requirements for a TES system in solar thermochemical processes: low cost of the materials, availability, high latent heat and high thermal conductivity, and an adequate melting temperature. This paper focused on the study of the metal alloys that can be used as a TES system, applied in the thermochemical process of endothermic chemical reaction for the production of solar fuel.

As described in the previous section, the steam reforming of natural gas occurs at temperatures higher than 650 °C, with the methane conversion levels of > 90% at an ambient temperature. The melting temperature of the selected material is therefore a key parameter to consider and should also be above 650 °C. Concerning the cost and

availability of the materials, magnesium (Mg), copper (Cu), silicon (Si), aluminum (Al), and some others were found to be the most appropriate.

Recently, most investigation was concentrated on aluminum eutectic alloys as PCMs in high temperature TES systems because of their suitable phase-change temperature, high latent heat density, and good thermal reliability [19]. In [18], the largest fusion heat, based on mass or volume, was obtained with binary and ternary alloys of the relatively plentiful elements Al, Cu, Mg, Si, and Zn. However, not all these metallic materials were suitable as PCMs for use in TES systems. In comparison with Na_2CO_3 molten salt, aluminum-silicon alloy (Al-Si alloy) as a PCM thermal storage medium has higher thermal conductivity at 190 [W/m K], larger latent heat and smaller thermal expansion, as shown in Figure 1. In this paper, therefore, the focus was on the potential of Al-Si alloy as a PCM thermal storage material.

Some advantages and disadvantages of the Al-Si alloy in comparison with the Na_2CO_3 molten salt for use in the reactor tubes were also listed in Figure 1. Noteworthy characteristics of the Al-Si alloy are as follows: (1) the temperature range for latent heat can vary depending on the Al-Si composition. The melting of eutectic Al-Si at 577 °C and the melting of the primary Si crystal above 577 °C can both be used for latent heat thermal storage. This means there is large latent heat capacity. (2) Thermal expansion can be controlled by the addition of silicon. Therefore, the volume change of the PCM can be suppressed in the transformation phase between the solid and the liquid phase. However, the disadvantages for the Al-Si alloy are high reactivity to stainless steel at high-temperatures and being susceptible to oxidization exposed to air.

The binary-phase diagram of the Al-Si alloy is shown in Figure 2. There are two primary solid solutions (the Al-rich phase containing 1.5at.%Si, and the Si-rich phase containing a maximum of 0.016at.%Al), the eutectic alloy (87.8wt%Al-12.2wt%Si), and no intermetallic phases in the binary-phase diagram [20]. The Al-rich side of the eutectic alloy was termed the “Hypoeutectic Al-Si Alloy”, with a composition of less than 12.6wt%Si in the two-phase coexistence region. The Si-rich side of the eutectic alloy was termed the “Hypereutectic Al-Si Alloy”, with a composition of more than 12.6wt%Si in the two-phase coexistence region. Concerning heat capacity, it is desirable that a hypereutectic Al-Si Alloy be used for sensible and latent heat TES, as is described below.

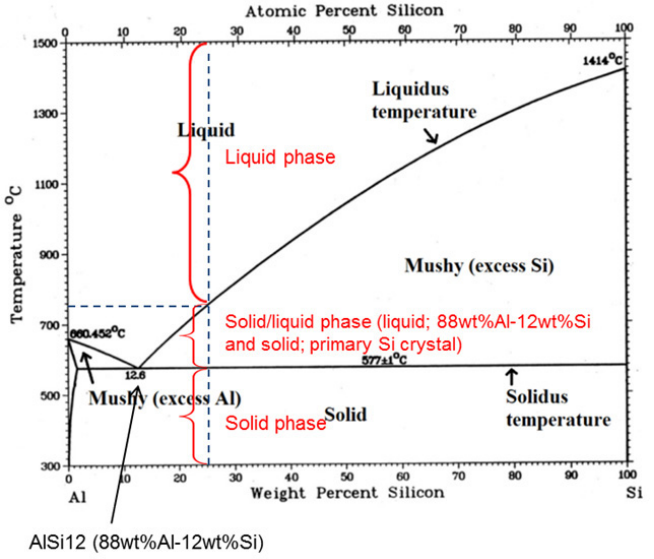

Figure. 2 Binary phase diagram of Aluminum and Silicon [20].

Figure 3 shows the theoretical heat storage capacities per unit volume and unit weight for the Al-Si alloy. Na_2CO_3 molten salt was also calculated and plotted as a reference in this figure. The value for heat storage capacities were calculated using the thermodynamic equilibrium software FactSage ver 6.4, and was plotted as a standard based on a temperature of 550 °C. Latent heat at a temperature of 577 °C and the sum of latent heat and sensible heat at 550-1000 °C are listed in Table 1. Concerning the heat storage capacities per unit volume, as shown in Figure 3, it is observed that latent heat is released by the eutectic alloy (87.8wt%Al-12.2wt%Si) at a temperature

of 577 °C for the Al-Si alloy. In a temperature region between 577 °C and the liquidus temperature, latent heat from primary Si and sensible heat from the eutectic alloy are contributed to increase the heat storage capacity of all the Al-Si alloys. Thus, the behavior in this range is sensible-like. Beyond the liquidus temperature, sensible heat from the liquid phase causes the heat storage capacity of all the Al-Si alloys to increase further. On the other hand, the heat storage capacity for the Na_2CO_3 molten salt is lower than for all the Al-Si alloys at temperatures of less than 858 °C, and is almost equal to the temperature of the Al-35wt%Si alloy. These results indicate that the latent heat of the eutectic compound and primary Si is available to use in the solar reforming of natural gas, as well as the Na_2CO_3 molten salt. Concerning the heat storage capacities per unit weight, the heat storage capacity for all the Al-Si alloys is higher than that for the Na_2CO_3 molten salt at temperatures below 800 °C, but at temperatures higher than 858 °C, the Na_2CO_3 molten salt has heat storage capacity levels of >1200 kJ/kg, which is higher than for all the Al-Si alloys. Here, in view of the limitations in useable volume for TES, a volumetric heat storage capacity should be preferentially evaluated for use in solar reforming and other thermochemical processes.

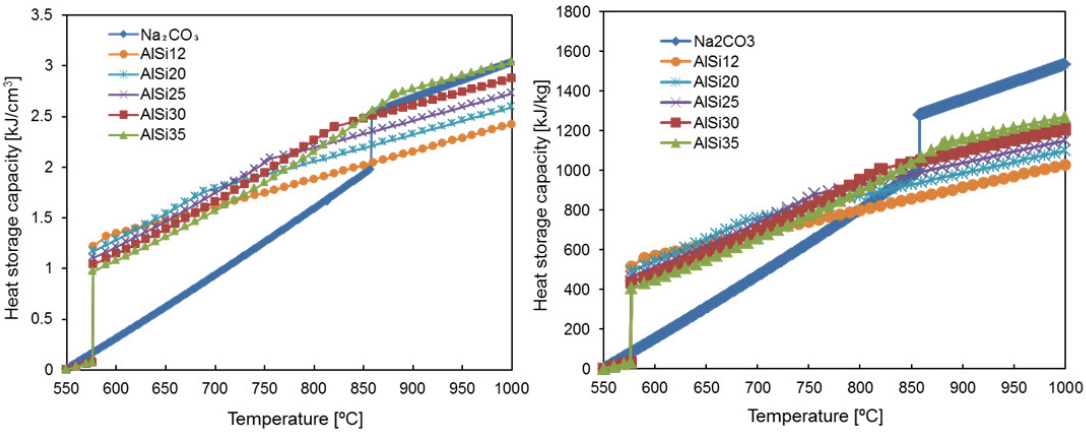

Figure. 3 Theoretical heat storage capacities per unit volume (cm^3) and unit weight (kg) for Al-Si alloys and Na_2CO_3 molten-salt. The heat storage capacity at temperature of 550 ºC as a standard is set to zero.

Table 1 Latent heat at temperature of 577 ºC and heat storage capacity at 550-1000 ºC for all Al-Si alloy and Na_2CO_3 molten salt thermal storages.

Storage Material	Heat storage capacity		
	Latent heat at 577 °C [kJ/kg]	Latent + sensible heat at 550-1000 °C [kJ/kg]	Latent + sensible heat at 550-1000 °C [kJ/cm^3]
AlSi12	483	1030	2.43
AlSi20	462	1100	2.59
AlSi25	434	1151	2.73
AlSi30	405	1207	2.88
AlSi35	376	1270	3.04
Na_2CO_3 (Molten salt)	—	1534	3.03

3. Cyclic test of thermal storage/discharge in vacuum for Al-Si alloys

Figure 4 shows the experimental setup for a cyclic test of the thermal storage/discharge in vacuum for the Al-Si alloy TES. The test container (length 200 mm, inner and outer diameters 93.6 mm and 101.6 mm, respectively) used for the cyclic testing of thermal storage/discharge was made from stainless steel (SUS310S). The PCM container (length 35 mm, inner diameter 30 mm, and thickness 10 mm) was made from graphite carbon and it was placed

inside the test container. The PCM container of the test container was filled with 29.5 g of Al-20wt%Si, 25.9 g of Al-25wt%Si, 30.1 g of Al-30wt%Si, and 28.5 g of the Al-35wt%Si alloy TES respectively as thermal storage. The Al-Si alloy, in the form of an ingot, was purchased from Nikkei Mc Aluminum Co., Ltd. The ingot was cut into a cylindrical shape, and was subsequently chemically washed and cleaned to remove oil and dust on the surface before being placed in the PCM container. The test container was vacuumed by connecting it to a rotary pump and was placed vertically in an electric furnace. The PCM container placed in the vacuuming test container was heated to 1200 °C maximum, with a heating rate of 5 °C/min to start the cyclic test of thermal storage/discharge. The test container was gradually heated, while keeping it in vacuum, for observation of the variations in the PCM temperature in order to evaluate the heat storage performance during heating (dT/dt versus heating time). After the temperature of the Al-Si alloy TES had risen above the liquidus temperature (the melting point of the Al-Si alloy), the temperature was kept constant for 1 h. Subsequently, the cylindrical furnace was switched off and the cooling of the test container started by natural convection. The test container was gradually cooled while keeping it in vacuum for observation of the variations in the PCM temperature in order to evaluate the heat-discharge performance during cooling (dT/dt versus cooling time). After the PCM temperature was below the eutectic temperature of 577 °C, the test container was reheated to 1200 °C. The periods of both heating and cooling varied depending on the chemical composition of the Al-Si alloy for 70-120 min. The PCM temperature was measured against time by a type K thermocouple, directly inserted into the graphite crucible inside the test container. These heating and cooling mode processes were repeated 20 times to evaluate the repeatability of the heat storage/discharge as latent TES and the compatibility with the PCM container. In order to evaluate the level of oxidation of the Al-Si alloy after the cyclic reaction (20 cycles), the Al-Si alloy TES was subjected to an electron probe microanalyzer (EPMA) to analyze each element.

The cyclic test for the thermal storage/discharge of the Al-Si alloy TES included two function modes: 1) a heating mode to simulate thermal storage (charging) by indirectly heating the thermal storage medium of the reactor by concentrated solar radiation; 2) a cooling mode to simulate thermal discharge from the Al-Si alloy TES to drive solar reforming during the passage of clouds.

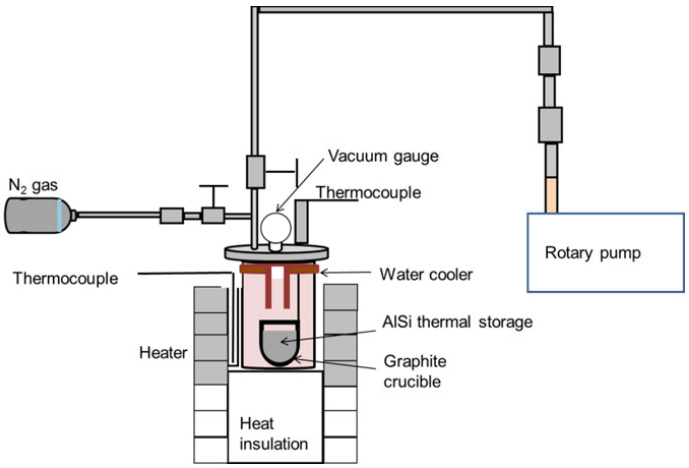

Figure 4. Experimental set-up for cyclic test of thermal storage/discharge in vacuum for Al-Si alloy TES.

4. Results and discussion

Figure 5 shows that the time derivative of the PCM temperature, dT/dt, was plotted against the PCM temperature for the Al-25wt%Si, Al-30wt%Si, and Al-35wt%Si alloys during the heat-discharge (cooling) mode. The cooling mode of the test container was performed by natural convection. As seen in Figure 5, exothermic peaks for Al-25wt%Si can be observed at temperatures of about 750 °C and 580 °C in the dT/dt profile. These peaks indicate the respective temperatures of the precipitation of primary Si during the solid to the liquid phase and the formation of the eutectic compound (87.8 wt%Al-12.2 wt%Si alloy) from the remaining liquid phase during the cooling mode of

the Al-Si alloy. These temperatures are in agreement with the liquidus and eutectic temperatures of the Al-25wt%Si in the Al-Si phase diagram, shown in Figure 2. The d*T*/d*t* profile indicates exothermic peaks for the Al-30wt%Si at temperatures of about 790 °C and 580 °C. These temperatures for the Al-30wt%Si are very close to the precipitation of primary Si solid and the formation of the eutectic compound respectively. Similarly, exothermic peaks for Al-35wt%Si are found at temperatures of about 880 °C and 580 °C in the d*T*/d*t* profile. This corresponds with the two kinds of solidification from the liquid phase during the cooling mode, as well as the Al-Si alloy described above.

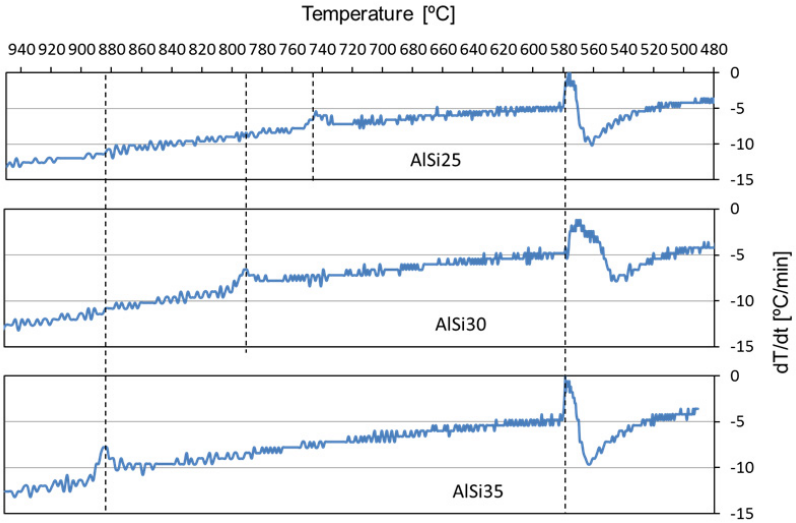

Figure 5. PCM temperature versus d*T*/d*t* for Al-25wt%Si, Al-30wt%Si and Al-35wt%Si alloy during the heat-discharge (cooling) mode.

Figure 6 shows the time variation for the PCM temperature and the time-derivative of the PCM temperature (d*T*/d*t*) for the Al-25wt%Si alloy. Concerning the heat storage (charging) mode, as seen in Figure 6(a), the rising temperature profiles of the Al-25wt%Si alloy were almost the same for the 1st, 10th, and the 20th cycle. The profiles of d*T*/d*t* decreased for all the cycles when the temperature was higher than 577 °C because of the latent heat storage by the melting of the eutectic compound at the eutectic temperature of 577 °C. The latent heat storage is therefore observed to be consistent (repeatable) during the course of the cyclic test. However, an obvious change of d*T*/d*t* is not observed at a temperature higher than 757 °C because of the latent heat storage by the melting of primary Si solid during the heat storage mode. For the heat discharging (cooling) mode, as seen in Figure 6(b), the temperature drop profiles of the liquid phase of the Al-25wt%Si alloy were observed to be consistent for the 1st, 10th, and the 20th cycles. Additionally, the profiles of d*T*/d*t* for all the cycles locally increased at temperatures of 757 °C and 577 °C because of the discharge of latent heat by the solidification of primary Si solid and the eutectic compound respectively. The results for Figure 6 indicate that the latent heat storage/discharge for the Al-25wt%Si alloy can be cyclically performed with good repeatability without the degradation of the performance in vacuum.

Figure 7 shows the results for the point analysis of the oxidation state of the Al-25wt%Si alloy. The Al-25wt%Si alloy before and after the 20th cyclic test was cut across, and the cross-section surface was analyzed by EPMA every 3 mm from the center position to the outer circumference. The oxygen fraction was plotted against the positions on the cross-section surface. The Al-25wt%Si alloy showed little evidence of oxidation at any position during the 20 cycles compared with before the cyclic test. In addition, the Al-25wt%Si alloy showed no reaction with a graphite crucible in vacuum during the cyclic test. Consequently, a graphite carbon may be used as a passivation layer in vacuum between a solar thermochemical reactor made from stainless steel and an Al-Si alloy thermal storage. Figure 8 shows a SEM photograph of the cross-section surface of the Al-25wt%Si alloy and element mapping by EPMA for aluminum, silicon, and oxygen after 20 cycles. In the SEM photograph, the observation of the white-colored primary Si crystals and the gray-colored eutectic compound is as good as before the cyclic test. In contrast with the SEM photograph, element mapping indicates that the primary Si crystal accounts for most of the silicon and the eutectic compound has a higher proportion of aluminum with a small fraction of silicon. Oxygen spread is

diluted, if at all present, for the aluminum region in the Al-25wt%Si alloy. The chemical composition measured by EPMA is an average of 75.13wt%Al-23.69wt%Si for the area surface of the Al-25wt%Si alloy.

5. Technical implementation

Figure 9 shows a conceptual diagram of the double-walled receiver/reactor system with high-temperature thermal storage of the Al-Si alloy combined with a tower-type solar concentrating system. Double-walled receiver/reactor tubes are installed in the concentrating area of the solar tower, and the concentrated solar radiation is converted to high-temperature solar heat. The solar heat can be stored as sensible and latent heat by the Al-Si alloy packed in the annular region between the inner tube and the outer wall of the receiver/reactor. If materials with large heat capacity, such as ceramic balls and honeycomb, are placed in the inner tube and air is passed through the inner tube, the double-walled receiver/reactor system will function as a solar receiver tube with the thermal storage of the Al-Si alloy intended for use in CSP. On the other hand, if catalysts for methane reforming are packed in the inner tubes and a gas mixture of methane and steam is passed through the inner tubes, the double-walled receiver/reactor system will function as a solar reformer, with the thermal storage of the Al-Si alloy intended for use in the production of solar thermochemical fuel. The system composed of the solar receiver/reactor together with the latent heat TES can continue to produce high-temperature air or synthetic gas ($CO + H_2$) without interrupting the gas supply if cloud passages occur for a limited time.

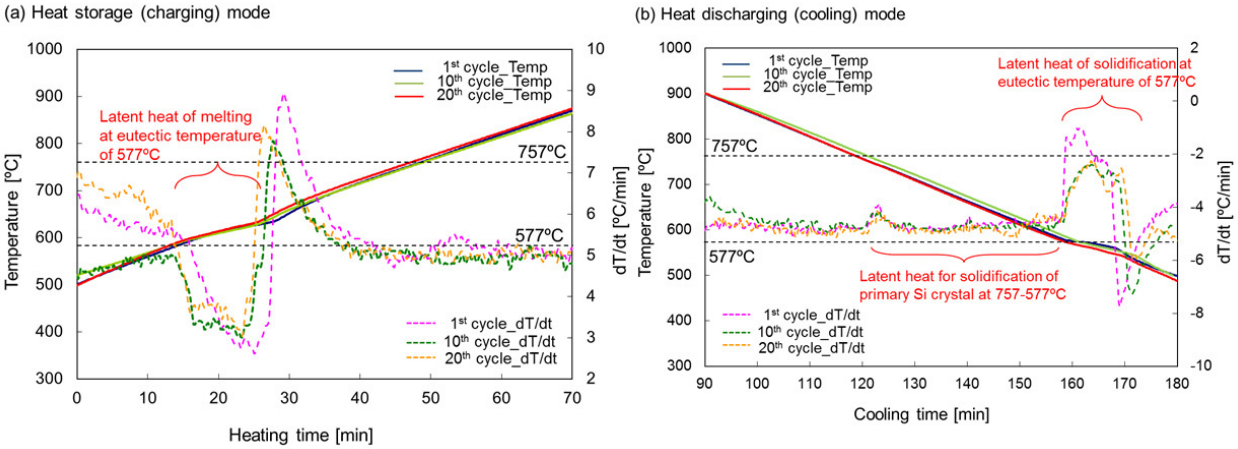

Figure 6. Time variation for PCM temperature and time-derivative of PCM temperature (dT/dt) for the Al-25wt%Si alloy: (a) heat storage (charging) mode; (b) heat discharge (cooling) mode.

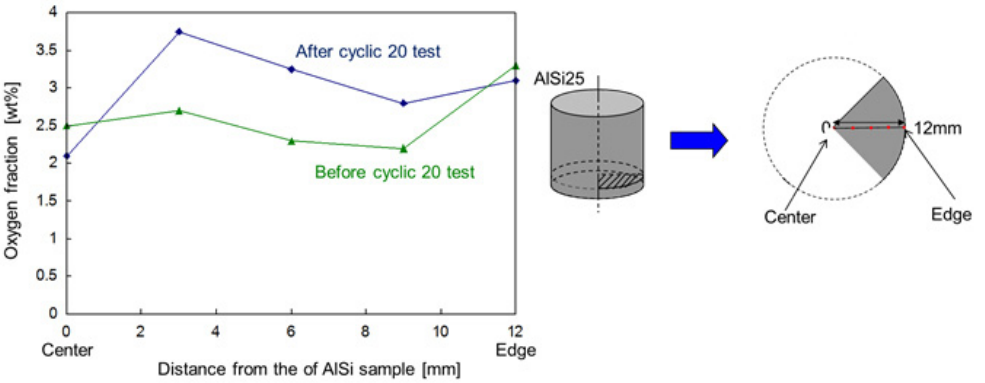

Figure 7. Analysis of oxidation analysis for the Al-25wt%Si alloy before and after 20th cycle tests. A point analysis was performed from the center cylindrical shaped-sample to outer circumference by EPMA.

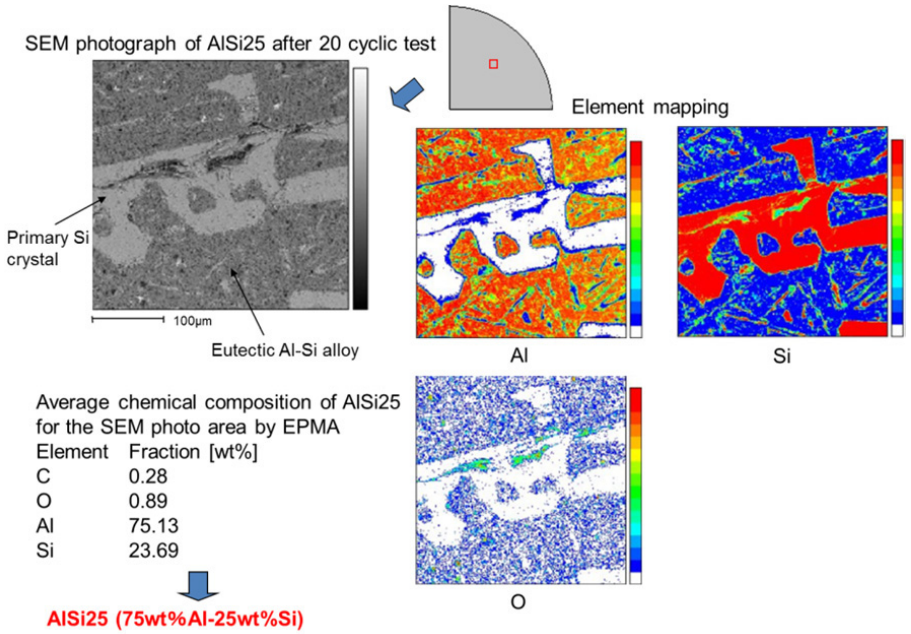

Figure 8. SEM photograph for cross-section surface of Al-25wt%Si alloy and element mapping by EPMA for Aluminum, Silicon and Oxygen after 20 cyclic test.

6. Summary

Al-Si alloy was studied as a new latent TES, alternative to the Na_2CO_3/MgO composite, for the cyclic properties of the thermal storage/discharge of the Al-Si alloy in vacuum to evaluate the alloy as a potential PCM thermal storage material. Latent heat from the eutectic alloy (87.8wt%Al-12.2wt%Si) at a temperature of 577 °C, latent heat from primary Si and sensible heat from the eutectic alloy in a temperature region between 577 °C and the liquidus temperature, and sensible heat from the liquid phase beyond the liquidus temperature causes the heat storage capacity of all the Al-Si alloys to increase, and have a potential for use in solar reforming and other thermochemical processes. Especially, concerning the heat storage capacities per unit volume, all the hypereutectic Al-Si alloys have higher heat storage capacities than the Na_2CO_3 molten salt at temperatures of less than 858 °C, and are almost equal to the Al-35wt%Si alloy.

The temperature increasing performance of the Al-Si alloy was measured during the thermal storage (heat-charge) mode and during the cooling (heat-discharge) mode. During heat-charging mode, a latent heat due to melting at eutectic temperature of 557ºC was observed. On the contrary, during heat-discharging mode, a latent heat at 557ºC and latent heat for solidification of primary Si crystal at 557-757 ºC was observed in the cyclic test through 20 cycles. Through the cyclic test of 20 cycle, Al-25wt%Si alloy was not oxidized, and reproducible charging/discharging of latent and sensible heat could be observed in this study.

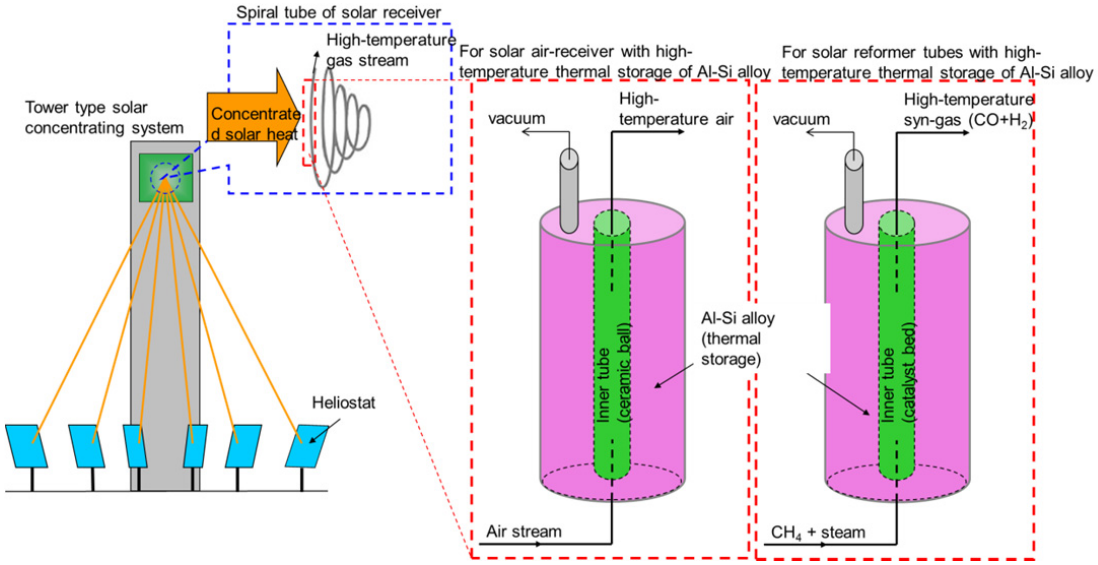

Figure 9. Conceptual diagram of double-walled receiver/reactor system with high-temperature thermal storage of Al-Si alloy combined with tower-type solar concentrating system.

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